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PATENT APPLICATION  
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**The United States Patent and Trademark Office**

In reapplication of: Paul Bruinsma, Suresh Baskaran, Jagannadha R. Bontha, Jun Liu

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Group Art Unit: 1755

For: MESOPOROUS-SILICA FILMS, FIBERS AND POWDERS BY EVAPORATION

Assistant Commissioner for Patents  
Washington, D.C. 20231

**DECLARATION OF DR. LIU  
UNDER 37 CFR § 1.132**

1. I am a Senior Staff Scientist (highest technical rank) at Battelle Memorial Institute (BMI), assignee of the above Bruinsma reissue patent application, and am one of the co-inventors of the invention described and claimed therein. My responsibilities include principal investigator and program manager for a wide range of programs for the Department of Energy, including one program related to the fundamental interactions between surfactants and metal oxide precursors in aqueous solutions. I have been a staff scientist with BMI for more than seven years.
2. My education includes a baccalaureate degree in Chemical Engineering from Hunan University and post-graduate degrees, including a Masters of Science in Ceramic Engineering and a PhD. in Materials Science from University of Washington. I have received the R&D 100 Award, Basic Energy Science Materials Science Award for Materials Chemistry: Significant Implication for DOE-Related Technologies, Best Paper Award of the American Ceramic Society, and Finalist for the Discover Magazine Award for Technology Innovation.
3. Previous work experience includes research assistant professor and research scientist at University of Washington from June 1990 to September 1992. My responsibilities included

performing research in ceramic synthesis, managing the Electron Microscopy Center, training students and faculties, and teaching electron microscopy.

4. I am a member of American Ceramic Society, American Chemical Society, and the Materials Research Society since 1990. I have published the following technical articles of interest in reputable professional journals, including Science, in connection with U.S. Patent No. 5,922,299 to Bruinsma et al. (hereinafter, the Bruinsma patent) (many of this articles are invited review articles, invited feature articles, or cover stories):

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- (13) B. J. Palmer and J. Liu, "Simulation of Self-Assembly in Surfactant Solutions," *Langmuir*, 12, 746-753 (1996).
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Microcavities in Nanoporous in Nanoporous Channel," hot paper, *Angewandte Chemie International Edition*, 39, 2702-2707, 2000.

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5. I have thoroughly studied and understand the Bruinsma patent and the patents and technical publications cited in the Background section thereof. I also have a working knowledge of the field of surfactant chemistry and the use of surfactants as templates to make mesoporous films and powders.

6. I have thoroughly studied and understand U.S. Patent No. 5,858,457 to Brinker, et al. (hereinafter the Brinker patent) and the patents and technical publications cited in the Background section thereof.

7. I am an expert in the field of surfactant chemistry and mesoporous materials. I have given invited seminars and plenary lectures in numerous international and national conferences and workshops, and in many major universities in the area of surfactant templated mesoporous materials. My work in this area has been widely reported and featured by Scientific American, Popular Science, Discover, Business Week, Popular Mechanics, and many other newspapers and magazines.

8. The term "critical micelle concentration (CMC)" as it appears in the Brinker patent refers to a threshold surfactant concentration beyond which the addition of new surfactant to the solution favors the formation of micelles (micelles are aggregated clusters of surfactant molecules), instead of free surfactant molecules. (See, for example, J. Israelachvili, *Intermolecular & Surface Forces*, Academic Press, San Diego p.351 (1992), attached hereto as Exhibit A.) This term of art would have been known by one of ordinary skill in the art at the application filing date of the Bruinsma patent, or August 26, 1997. At the CMC, several thermodynamic

properties, such as conductivity, surface tension, turbidity, and osmotic pressure go through a discontinuous transition. I and my colleague Dr. Liang Liang, a research scientist with BMI working under my supervision, have replicated experiments in Example 1 of the Bruinsma Patent in order to confirm that the precursor solution used in the original experiments inherently included micelle concentrations much less than the critical micelle concentration to which Brinker alludes.

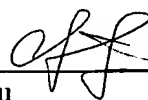
9. I and Dr. Liang Liang replicated the experiments by carefully replicating and controlling the conditions, the chemicals, the process steps, and the CTAC/TEOS mole ratios described in the reissue application with a molar ratio for TEOS:H<sub>2</sub>O:HCl:EtOH of 1.0:7.2:0.1:5.7 that was used in Example 1 to generate Figure 2 in the Bruinsma patent. The surfactant concentration was varied to cover all the data points in Figure 2. We then determined the CMC, by measuring the conductivity of the solution as a function of the surfactant concentration, as would have one of ordinary skill in the art who had read the Brinker patent and its claims containing that term. The CMC for the solutions used in Example 1 for Figure 2 were determined to be about 0.21 mol/liter. This value translates to a CTAC/TEOS ratio of about 0.16. I have shown the position of the CMC on the graph of Fig. 2 of the Bruinsma patent, attached hereto as Exhibit B. It may be seen from this plot that the surfactant concentrations of many of the experimental results (i.e., those having a CTAC/TEOS MOLE RATIO of less than approximately 0.16), are below the CMC and a few are above the CMC. Of those results below the critical micelle concentration, it is my belief that at least three (i.e., those having a CTAC/TEOS MOLE RATIO of less than approximately 0.10) are "much less than" the CMC, as Brinker used that term, for example, in claim 1. I believe that because of the definition of CMC, one of ordinary skill would have concluded that "much less than" would include surfactant concentrations representing less than a substantial fraction of the CMC (i.e. surfactant concentrations sufficiently below the CMC to provide a margin of error not to exceed the CMC), certainly those that are less than a CTAC/TEOS ratio of 0.1 in Figure 2.

10. In a brief summary, it is my expert opinion that a micelle concentration much less than a CMC would have been concluded by a person of ordinary skill in the pertinent art on August 26,

1997 to be an inherent characteristic of at least some of the precursor solutions described and illustrated in the Bruinsma patent.

11. I, the undersigned, declare that all statements made herein of my own knowledge are true, and that all statements made on information and belief are believed to be true; and further, that these statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Dated this 7th day of November, 2000.

  
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Jun Liu  
Senior Staff Scientist (Grade VI)  
of Battelle Memorial Institute, Richland, WA